EXTRACTION, CHARACTERIZATION AND FUNCTIONAL PROPERTIES OF NEWLY IDENTIFIED ACACIA CAESIA PLANT FIBER REINFORCED EPOXY POLYMER COMPOSITES

A SYNOPSIS

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1. INTRODUCTION

Composites, plastic materials, and ceramics have dominated the emerging material landscape for three decades. Composite materials have been steadily increasing in volume and number of applications, gaining a foothold in these, and conquering new markets. There are many modern composite materials today, ranging from everyday products to high-tech niche applications. Fiber glass-reinforced plastics (FRP) are now used in a wide range of products, from composite armouring to natural gas vehicle fuel cylinders, windmill blades, industrial drive shafts, bridge support beams, and even paper mill rollers. Composites rather than metals have resulted in cost and weight savings for specific applications. Furthermore, the need for composite materials that are lighter and more earthquake-resistant has put a lot of emphasis on using new and better materials that reduce the weight and absorb the shock and vibration through tailored microstructures, which are unique and offer improved properties. Depending on the type of reinforcement used, the final product's characteristics can be customised to meet nearly any engineering need. It is possible to get the best results by combining traditional materials with composites. To hasten the switch from synthetic to natural fibers in the textile industry, more natural fibers must be made available. In order to meet current requirement for these fibers, current production levels are insufficient. New plants must be employed for fiber production, especially in local contexts, able to allow for simple and inexpensive extraction methods without compromising the fiber's quality. The analysis of these new fibers is necessary to establish their morphological, physical, chemical, and mechanical properties. In particular, microstructural studies (whether using optical or scanning electron microscopy) can be of great assistance to better elucidate how these fibers look in practice.

Polymer Matrix Composites (PMC) are materials using organic polymers as matrix and fibre as reinforcement. There are different types of PMC according to the polymer types like thermoset and thermoplastic polymers. A resin will be therefore blended with fiber reinforcements to make polymer composites, which will be tested for their combined properties. Following this, a thorough understanding of how these fibers contribute to the composite's improved performance is essential.

In particular, Acacia caesia is famous for its medical value, since its leaves are used for the treatment of asthma, and skin diseases. The wood is used for making furniture, boats, and so forth. Acacia caesia bark (soap bark), referred to as Kari Indu, Indumul Kodi (Tamil), Incha, Palinja (Malayalam), can be considered as an agrowaste after its application as a natural body scrubber or after some ayurvedic treatment.

The fact that Acacia caesia bark has been known for a long time does not imply that its fibers are used in high-performance commercial applications, even though they are potentially available after bark extraction all over the world and in tropical, subtropical, and warm temperate areas. Exploratory research has been therefore performed on a new fiber from the Acacia caesia plant and the relevant composites obtained with epoxy matrix.

2. RESEARCH DISCUSSION

The research work concerned the possible use of Acacia Caesia bark (ACB) fiber, a typical residue of other processes, in particular in the traditional medicine and cosmetic field, for the prospective production of composites.

The work was then divided into two principal themes, namely the extraction and characterization of ACB fibers, and the production and characterization of epoxy matrix composites including different amounts (from 10 to 35%) of ACB fibers of different lengths from 10 to 30 mm.

3. LITERATURE REVIEW

This literature review gives the background information on the issues are to be considered in this thesis and to emphasize the relevance of the present work.

Gopinath et al. [1] investigated physico-chemical, mechanical, thermal, as well as morphological characteristics of a novel cellulosic fiber extracted from the bark of Bauhinia purpurea using chemical, tensile, Fourier Transform Infrared Spectroscopy, X-ray diffraction, TGA, DSC, and FESEM. Testing revealed that Bauhinia purpurea fiber (BPF) had a high cellulose content (67.46 wt.%), a high crystallinity index (65.61%), a high tensile strength (375.77-793.25 MPa), and a high Young's modulus (12.78-20.12 GPa). Because of its high initiation temperature of thermal breakdown (225.55 °C) and low density (1190 kg/m^3), BPF is better suited to producing biodegradable composites for use in the automotive and infrastructure industries.

Kumar et al. [2] studied the basic properties of a new plant fiber made from the bark of the Acacia nilotica L. tree. The chemical analysis was used to determine the different chemical components of the Acacia nilotica L. fiber (ANF), such as the percentages of cellulose (56.46 wt.%), hemicelluloses (14.14 wt.%), lignin (8.33 wt.%), and ash content (4.95 wt.%). The thermogravimetric analysis found that the highest temperature at which ANF degrades is 339°C. Based on the X-ray diffraction examination, ANF had a crystallinity index of 44.82%

and a crystalline size of 3.21 nm. Atomic force microscopy was used to approximate the topography of ANF's surface. The density of ANF was identified as a value of 1165 kg/m^3, which is considerably lower than the other fibers such as Acacia leucophloea (1385 kg/m^3) and Jute (1460 kg/m^3).

Kathirselvam et al. [3] in their study reported the effect of treating Thespesia populnea bark fiber with NaOH treatment in terms of surface and structural characteristics. This fiber had a reasonably high percentage of cellulose (76.42%). Fibers treated with 5% NaOH for 60 minutes showed a significant mechanical strength (678.41 ± 48.91 MPa). The elimination of non-cellulosic chemicals can be observed through Fourier transform infrared spectra, and X-ray diffraction research showed a 13.6% expansion in the size of cellulose crystals on optimally treated fibers. The dependability of the obtained tensile test results was statistically interpreted using the Weibull distribution model. In the end, microscopic studies using scanning electron microscopy and atomic force microscopy showed an increased roughness of the fiber surface after being treated with alkali, which provides a stronger grip for making them suitable for the reinforcement in polymer matrices.

Venugopal and Booninathan [4] studied the suitability of Acacia Concinna fiber (ACF) as the reinforcement for environmentally friendly composites by studying its basic features (density, diameter, chemical composition, crystalline nature, thermal behavior, tensile properties, and surface topography). The chemical analysis results showed that the most promising change in chemical composition occurred after 45 minutes of treatment with 5% (w/v) NaOH against other possible times, which were set at 15, 30, 60, and 75 minutes. The FTIR and NMR spectrograms showed that the amount of lignin and hemicellulose in the alkalized ACF decreased. X-ray diffraction (XRD) examination showed that when ACF was appropriately alkalized, its crystallographic characteristics (crystallinity index) increased from 27.5 to 35.6%, and its crystalline size decreased from 4.17 to 6.43 nm. A thermal analyzer is used to determine the mass loss pattern (TG & DTG profile) of both untreated & optimally alkalized ACF. The effect of alkalization on the ACF's surface topography was recorded using scanning electron microscopy and atomic force microscopy.

Muthalagu et al. [5] studied the tensile, flexural, impact, and hardness characteristics of *Prosopis juliflora* bark (PJb), jute fiber (JF), and kenaf fiber (KF) reinforced polyester composites for the first time. Natural fiber composites (NFC) provide a unique set of difficulties due to the wide range of their individual qualities and features. While the proportion of JF was kept constant to a 50% of the total fibers, the percentages of the other two fiber fillers were used in different proportions (40/10, 30/20, 25/25, 20/30, and 10/40). Tensile properties,

flexural properties, impact strength, and hardness tests were carried out according to ASTM standards to examine the material's mechanical properties. Mechanical tests showed that adding natural fiber fillers reliably increased the aforementioned mechanical benefits. The chemical composition of NF is determined using scanning electron microscopy (SEM) and Fourier transform infrared spectroscopy (FTIR) analyses, respectively. Thermogravimetric analysis was used to detect NF's thermal consistency/degradation (TGA).

Ramesh et al. [6] presented an experimental study on using *Calotropis gigantea* fiber (CGF) as reinforcement for constructing the composite material. The composite laminates were manufactured using a compression moulding technique and tested for water absorption and mechanical properties, including flexural strength, tensile strength, and impact resistance. According to the findings, CGF composites had the highest flexural strength of 87.88 MPa, a tensile strength of 62.72 MPa, and impact strength of 10.4 kJ/m^2, respectively compared to coir and cotton fiber reinforced composites. The water absorption and mechanical parameters of the CGF reinforced composites were comparable to those of other natural fiber reinforced composites explored in previous literature investigations.

Nayak et al. [7] examined the Ceiba pentandra (CP) fibers, which have been selected as the reinforcement for thermoplastic polymers, because of their low weight, high strength, and recyclable properties. When fiber loading is increased initially, mechanical characteristics improve up to the optimal level (20 wt. %) and then decrease. The mechanical parameters at the optimal fiber loading are a flexural strength of 98.49 MPa, impact strength of 32.01 kJ/m2, and a tensile strength of 85.92 MPa. Scanning electron microscopy, dynamic mechanical analysis, and thermogravimetric analysis were all used to learn about composites. TGA confirmed that the composites' thermal stability was improved above that of the plain polyvinyl alcohol (PVA) matrix.

4. RESEARCH GAPS

Presently a wealth of research works exists and are developed on some natural fibers like Jute, Flax, Hemp, Kenaf, Sisal, mainly extracted from the bast or the leaves of the plant. This is not the case with bark fibers, which are very diffuse, yet not very investigated. However, the nature of the plant, its place of cultivation, plant age and the method of extraction depends mainly decides on the properties of natural fibers. Acacia Caesia is an evergreen plant that has been identified as a major fiber source for ayurvedic use, especially for bath scribers. In South India, Acacia Caesia plant can widely be found to be grown, particularly in Kerala, some places

in Tamilnadu. ACBFs (Acacia Caesia Bark Fibers) in polymer-based epoxy matrices have not been used so far as reinforcement. With wide availability of ACBFs a novel attempt is made to conduct comprehensive investigations and to determine the different characteristics of ACBFs and the fabrication of composites also. The present work deals with the characterization and fabrication of Acacia Caesia Bark Fibers reinforced with Epoxy-based polymer composites.

5. RESEARCH OBJECTIVE

There are a number of primary goals for which this investigation is carried out:

1. Develop a procedure for extraction of fiber from the bark of the Acacia Caesia plant.
2. Explore physical, chemical, morphological, thermal, and mechanical properties of Acacia Caesia bark fibers, raw and treated with alkali tenors of 5, 10, and 15%.
3. Carry out mechanical and thermal characterisation of epoxy matrix based composite materials and to find the effect on raw fiber reinforcement using varying fiber lengths (10, 20, and 30 mm) and weight percentage (10, 15, 20, 25, 30, and 35 %).
4. Conduct fractographic analysis using Scanning Electron Microscopy of fractured specimens after various tests (Tensile and Izod impact).
5. Conduct the thermal characterization from composite's analysis of selected specimens (least (10%), average (25%), highest (35%)) after the TGA and DSC test.

6. METHODOLOGY

Describes the complete workflow of research methodology of the present investigation of ACBFs as shown in Figure 1.

7. RESULT AND DISCUSSION

7.1. Fiber characterization

7.1.1. Density and diameter measurements

Before the tensile test, density and diameter were measured to maintain specimen properties for analysis. Alkaline treatment removes amorphous components and reduces hemicellulose to improve fiber-matrix bonding, where 5% NaOH was applied for 60 minutes to improve composites. Standard test methods measured ACBF composition by cellulose, hemicellulose, lignin, moisture and ash content. The dry weight method for cellulose, hemicellulose and Klason lignin was used [8]. In contrast, the ACBF ash content has been

analyzed by the E-830 ASTM standard, and a method described elsewhere for moisture content, based on ASTM E-871 standard, has been used [9], as shown in Table 1.

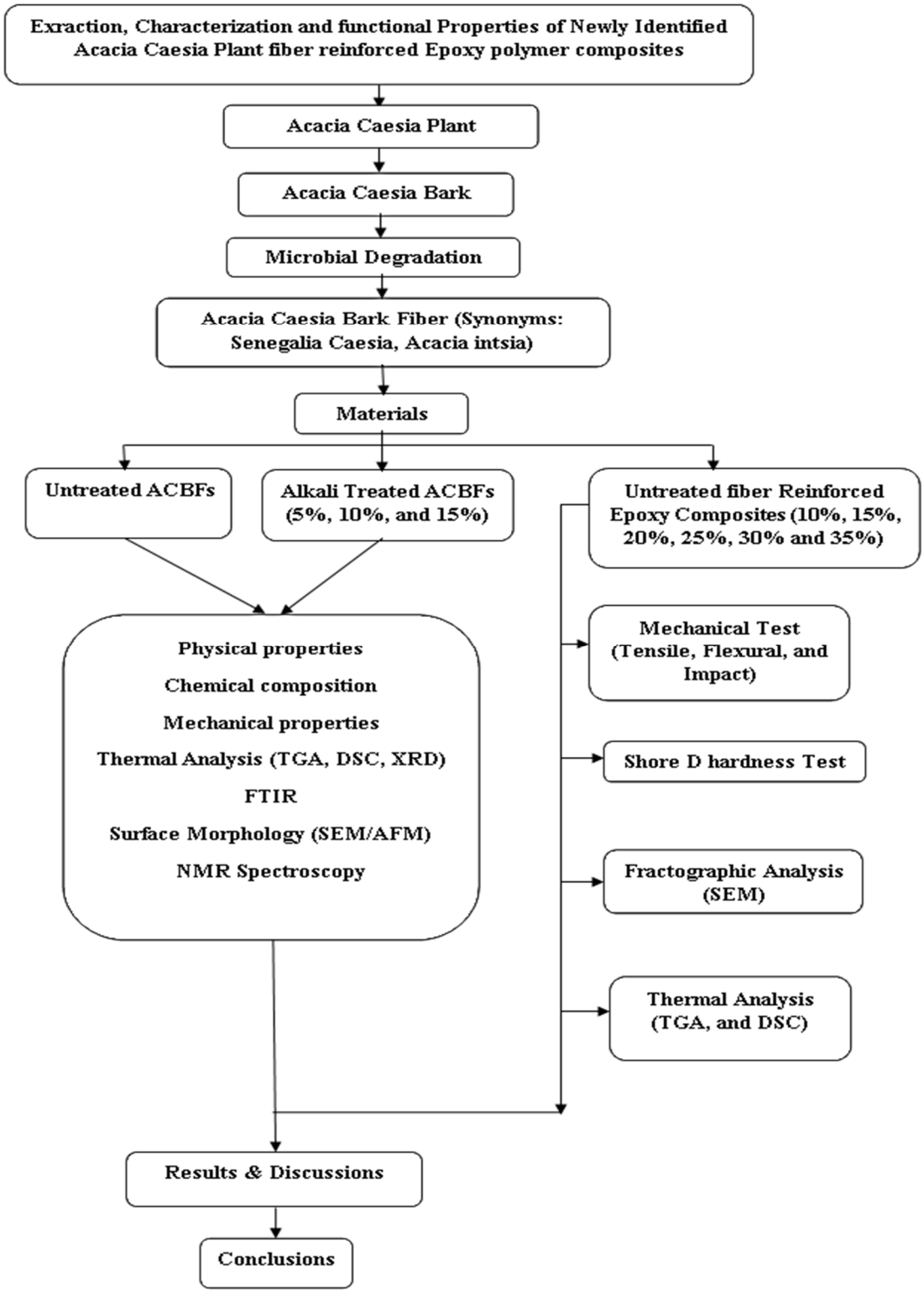
Exraction, Characterization and functional Properties of Newly Identified Acacia Caesia Plant fiber reinforced Epoxy polymer composites
Acacia Caesia Plant
Acacia Caesia Bark
Microbial Degradation
Acacia Caesia Bark Fiber (Synonyms: Senegalia Caesia, Acacia intsia)
Materials
Untreated ACBFs
Alkali Treated ACBFs (5%, 10%, and 15%)
Untreated fiber Reinforced Epoxy Composites (10%, 15%, 20%, 25%, 30% and 35%)
Physical properties
Chemical composition
Mechanical properties
Thermal Analysis (TGA, DSC, XRD)
FTIR
Surface Morphology (SEM/AFM)
NMR Spectroscopy
Mechanical Test (Tensile, Flexural, and Impact)
Shore D hardness Test
Fractographic Analysis (SEM)
Thermal Analysis (TGA, and DSC)
Results & Discussions
Conclusions

Figure 1 Research Methodology for Acacia caesia bark fiber epoxy Composite

Table 1. Physical and chemical properties of raw and alkali treated ACB fibers

Fiber type	Density (kg/m^3)	Diameter (µm)	Cellulose (%)	Hemicell. (%)	Lignin (%)	Ash (%)	Moisture (%)	Wax (%)	Extractives (%)
UACBF	1200	127±25	37	20	18	4.3	11.7	0.4	8.6
5% A-ACBF	1150	123±22	40.1	17.3	23.5	5.4	11.5	0.2	2
10% A-ACBF	1180	115±6	42.3	14.2	26	4.5	11.3	0.14	1.56
15% A-ACBF	1260	83±6	44.1	10.2	28	5.4	10.4	0.1	1.8

7.1.2. Fourier Transform Infrared Spectroscopy (FTIR)

Figure 2 shows untreated and treated ACBF (5%, 10%, 15% NaOH) FT-IR. Fibres are 4000–400 cm-1 functional groups. Hydrogen bond hydroxyl group O-H stretching peaks at 3874.34, 3945.63, 3958.47, and 3968.42 cm-1 for UACBF, 5%, 10%, and 15% AACBF [10]. UACBF, 5, 10, and 15% AACBF stretch cellulose-OH/-CH at 3280.49, 3390.26, 3378.41, and 3392.63 cm-1[11]. UACBF cellulose's C-H stretching vibration peaks at 2900.63 cm-1, 2925.85 in 5%, 2888.65 in 10%, and 2885.89 in 15% [12]. The carbonyl C=O linkage of the stretching vibration of the carboxylic acid in lignin or the ester group in hemicellulose produces peaks at 1613.78 cm-1 in UACBF, 1626.96 in 5% AACBF, 1624.18 in 10%, and 1620.52 in 15% [13]. The C-O stretching vibration of the acetyl group in hemicellulose and lignin correlates with absorbance maxima at 1317.27 cm-1 in UACBF, 1320.71 in 5% AACBF, 1314.92 in 10%, and 1310.27 in 15% [4]. Alkali degraded lignin, wax, hemicellulose, and other fibre functional groups [14]. Treated fibers have more cellulose than untreated ones, which may explain different absorbency peaks.

7.1.3. Scanning Electron Microscopy (SEM/AFM)

This morphological study obtained high-resolution SEM images from gold-sputtered samples. SEM micrographs of fibre surfaces show that extreme untreated and 15% treated fibres expose more cellular material (Figure 3a). The former may be due to more non-structural material, while the latter may be due to excessive removal. 5% and 10% treatment may smooth fibres. Treatment can change the geometry of flattened fibres. The atomic force microscopy (AFM) images of Acacia caesia bark fiber in 2D and 3D. AFM (Figure 3b) is used to measure the roughness of a fiber's surface in two- and three-dimensional topographical

textures, allowing for easy identification of the fiber's smoothness or conversely waviness. Finally, it is important to conclude that the alkali treatment smoothes out the roughness of the acacia caesia bark fiber by dissolving the protective hemicellulose and lignin structure as well as the wax layer

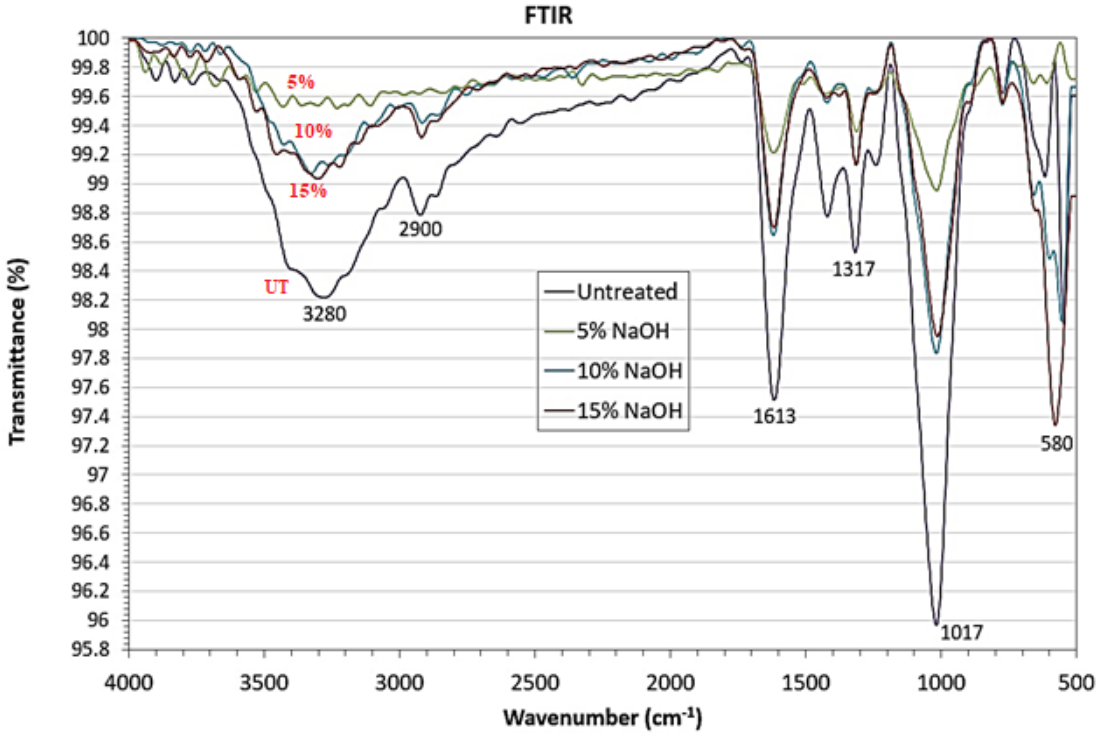

Figure 2. FTIR spectra of raw and alkali treated ACB fibers

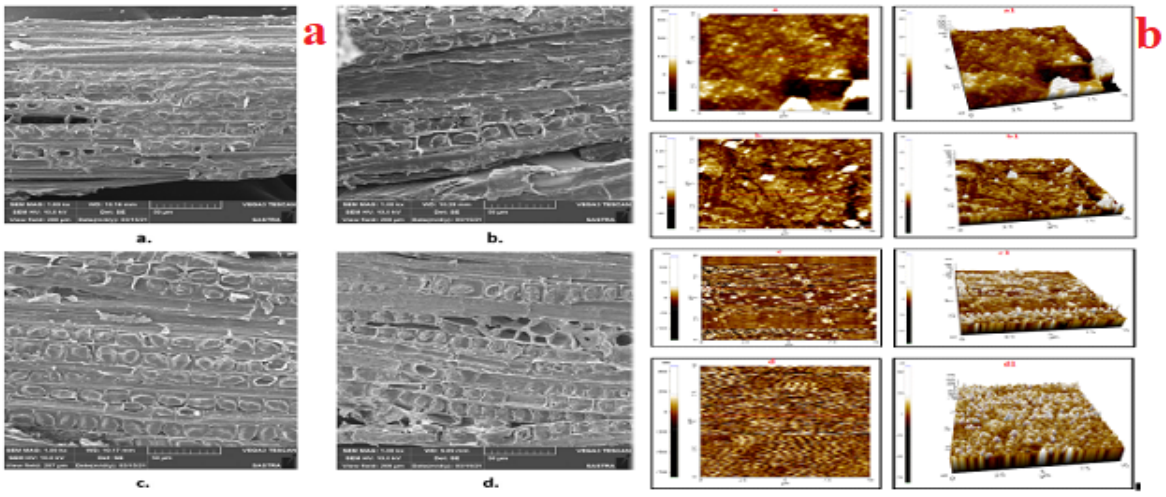

Figure 3. SEM (a) & AFM (b) images of raw and alkali treated ACBF

7.1.4. Thermal Analysis (TGA/XRD)

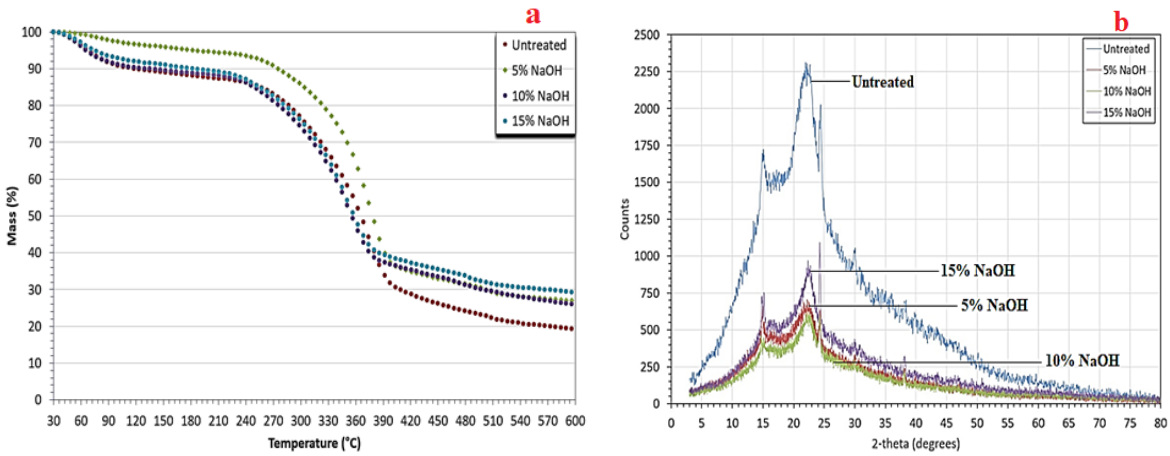

Figure 4. TGA (a) and XRD analysis (b) of raw and alkali treated ACB fiber

The thermal stability (Figure 4a) of the Acacia caesia bark fibre that was treated with alkali was improved in comparison to the untreated fibre, since the amount of non-structural and amorphous hemicellulose is gradually wiped out with it. This could be because of the char's increased rate of free radical formation, which is stabilised by the char's condensed carbon ring formations [15–17]. In practice, when the concentration of the alkali was increased from 5 to 15% NaOH, the residual char content at 500°C increased from 4.87 to 24.41%. From XRD diffractograms of untreated and alkali-treated ACB fibers (Figure 4b), the fibers exhibit two prominent peaks at 2θ values at 15.23 and 22.21°.The peak at 15.23° was of lower intensity representing amorphous material (I_{am}), while the peak at 22.21° indicates the availability of crystalline cellulose in the fiber (I_{002}). In contrast, the peak at 2θ = 18.50° revealed the occurrence of non-cellulosic materials, such as hemicellulose, amorphous cellulose, pectin, and lignin in the fiber. It can be stated that the treatment does not remove any amorphous cellulose where as lignin and other non cellulose parts are removed. The crystallinity (25.10%, 22.53%, 24.09%, and 22.68%) of the acacia caesia bark fibers decreases as the alkali concentration increased from 5% to 15%.

7.2 Mechanical Properties

The following figures explains the research methodology of Acacia caesia bark fiber characterization, reinforced epoxy composite materials as well. The mechanical strength

(tensile, flexural, and impact) and shore D hardness of the ACBF-epoxy composites with respect to different fibre lengths and wt% are presented in Figure 5.

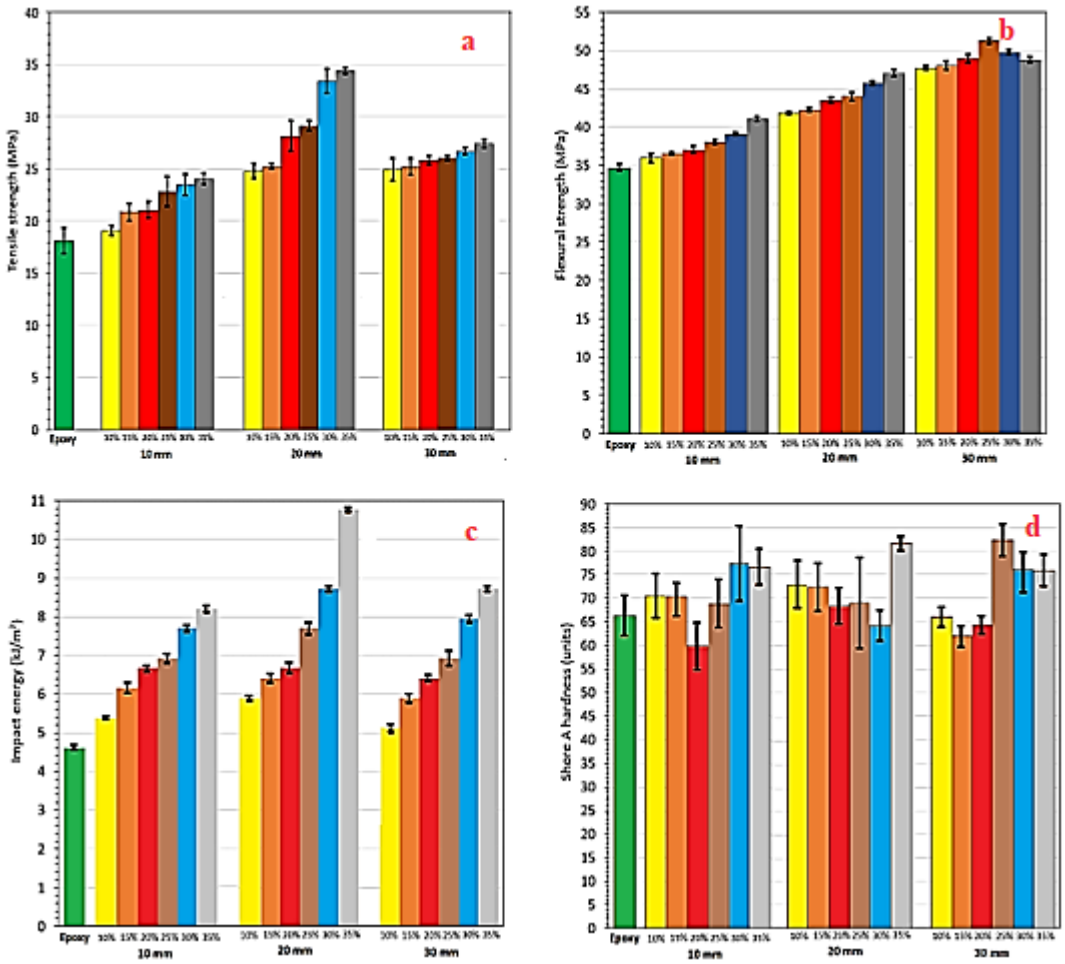

Figure 5 Mechanical strength of ACBF composites fabricated with different fiber lengths and wt.% ; a) Tensile strength, b) Flexural strength, c) Impact strength, d) Hardness (Shore D)

Figure 5a shows that it has been observed that the tensile strength of the composites increases the higher the fiber length and the fiber content introduced. The composites reinforced with 10 mm fiber and 10 wt% were observed to have the lowest value of tensile strength, compared to other composites. The increase in tensile strength of the composites with at higher wt% is attributed to increased interfacial strength between fiber and matrix due to higher surface area exposed to matrix, and also richer fiber wetting with epoxy matrix resulting in greater fiber-matrix adhesion. In this study, the maximum tensile strength of 34.41 MPa was obtained when introducing 35% fibers 20 mm long.

From Figures 5b (flexural strength) revealed also that the flexural properties of acacia caesia bark fiber reinforced epoxy composite are generally higher than those of pure epoxy.

Moreover, the composites including 25 wt.% of 30 mm long fibers are those showing the maximum flexural strength, equal to 51.21 MPa. This is attributed to the stronger bonding of fiber and matrix reported to that level of reinforcement and to that fiber length. It is suggested therefore that under flexural loading the non uniformity of ACB fibers constitutes a lower limitation than for tensile loading, therefore also a 30 mm long fibers would be suitable for use in the composite.

Figure 5c shows that, The effect of fiber loading on the impact strength of the Acacia Caesia Bark fiber composites. The figure indicates that impact strength is increasing with the introduction of more fibers in the composite. The impact strength of composites with 35 wt.% 20 mm fiber long fibers was found to reach the maximum value of 10.76 kJ/m^2, when compared to all the other composites. At lower fiber loading, the impact strength is less, possibly due to the presence of critical defects, such as voids, and improper bonding at the interfacial regions, as typical for limited concentration on bark fibers in composites [18]. As the fiber loading is increased gradually, the reinforcing nature of cellulose present in the fiber neutralized the defects and transmitted improved toughness [19].

Hardness values for the produced composites (Pure Epoxy, Epoxy + ACB with varied fiber length and wt.% fraction) (Figure 5d), it can be observed that, increasing the wt.% and length of acacia caesia bark fiber resulted in a noticeable effect on hardness of the material. It is concluded that the presence of more abundant and longer fibers in the composites, by enhancing the elasticity, does also improve the surface resistance to indentation, yet also induces some susceptibility to the state of the surface, roughened by the presence of more fibers.

7.3 Morphological study by Scanning Electron Microscopy

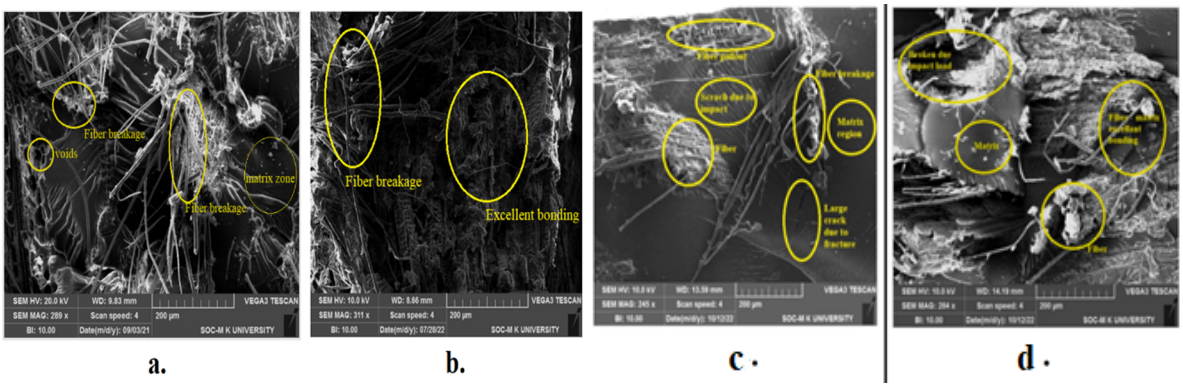

Figure 6. (a) 10 mm 10% tensile, (b) 10 mm 35% tensile, (a) 30mm 10% impact, (b) 30mm 35% impact

Figure 6 (a) explores 10 mm 10% composites after tensile testing. It can be seen from the figure that there are places of voids due to air traps [20]. It is also evident that some segment of fibers buckle into microfibrils during breakage. The failure seen is mostly due to failure of matrix after the fiber failure, since more of matrix phase is visible. Figure 6 (b) shows the tensile fractograph of 10 mm 35 wt.% composite that had maximum tensile strength. The increased tensile strength may be attributed to the formation of effective bonding and more fiber occupying the available space compared to 10 wt % composite.

From Figure 6 (c) and 6 (d) Fiber pullout in the case of composites with 30 mm and 35 wt% is further reduced compared to that of 10 mm 35 wt% and 20 mm 35 wt% composites. Good fiber matrix adhesion was seen which has led to the possibility of formation of matrix cracking and matrix shear. Less fiber pullout and reduced matrix cracking contributed much for the improved properties observed for the 30 mm and 35 wt% composites. However, the 20 mm 35 wt% composites have equally similar behaviour in terms of the fracture mechanism but the optimal fiber length, which has enabled a good reinforcement effect has resulted in the improved properties in the case of 30 mm 35 wt% composites.

7.4 Thermal Study of ACBF-Epoxy Composites

The TGA/DSC curves for the composites with varying fiber length and wt% is shown in Figure 7 (a) and 7 (b). It is evident from the figure that the composites with reinforcement are having improved thermal stability and highest heat flow compared to that of the pure resin, in all cases the curves being lower than that of the pure epoxy. In general, the thermal stability is found to be enhanced on increasing the wt% of fibers. As far as the thermal stability of the 20 mm 35 wt% composites is concerned, they exhibited good thermal resistance to degradation compared among all the other composites. The length of fiber is also an important aspect related to the behaviour of the composites under change on temperature. The composite with those two design characteristics (amount and length of fibers) exhibited improved thermal stability compared to the others tested in the investigation.The inclusion of the fibers in the composite did possibly also enhance their contribution into thermal behaviour, showing the typical peaks between 375 and 460 °C, as reported also e.g., in a very recent work on babassu fibers, beyond the typical peaks to be attributed to moisture dissolution and hemicellulose degradation, at around 60 and 140 °C, the latter not much evident into the composite.

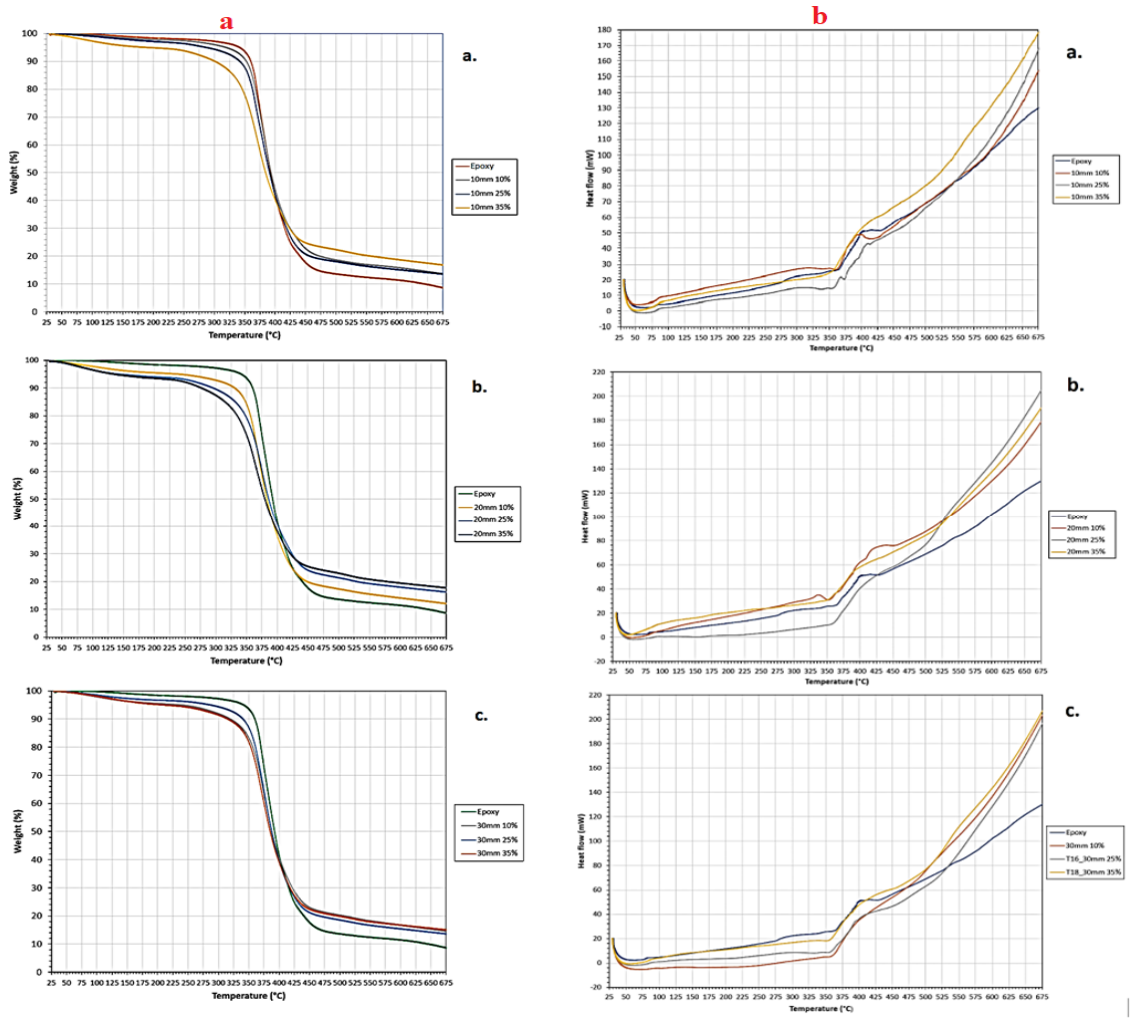

Figure 7 Thermal properties of ACBF composites fabricated with different fiber lengths and wt.%; (a) TGA, (b) DSC

8. ORGANIZATION OF THESIS

- **Chapter 1: Introduction**

This Chapter provides an overall view on composite materials, classifications, advantages, disadvantages, and applications. It also briefs about polymer matrix composites with many types of natural fibres as reinforcement and main types of thermosetting resins (unsaturated polyester, epoxy, vinyl ester) as the matrix. The discussion also explains the acacia caesia bark fiber used as reinforcement as well as epoxy resin as matrix to prepare the fiber characterizations, composites fabrication and mechanical and thermal characterization.

- **Chapter 2: Literature Review**

This Chapter provides the information about the recent literature review on different types of natural fibres characterizations (physical, chemical, mechanical, morphological, and thermal). The study extends to select a suitable fabrication technique, effect of fabrication conditions, process parameters and its influence on their mechanical, morphological and thermal characteristics. Based on the literature review, it presented the knowledge gap in this area. The objectives of this research work are also presented.

- **Chapter 3: Materials and Methods**

This Chapter provides the research methodology used for this research work. The properties of matrix and reinforcement materials are explained in detail. Different chemical treatment with raw fiber characterizations and then required number of composite plates were made by using compression moulding techniques with varying fiber length and weight percentage of the materials. The equipments used for the characterization studies of Acacia caesia bark (ACB) fibers, composite plates mechanical and thermal characterizations to measure the quality characteristics of machined composites are also discussed in detail.

- **Chapter 4: Characterization of raw and alkali-treated ACB fibers**

This Chapter describes the characterization of raw and alkali acacia caesia bark fiber using physical property analysis, chemical property analysis, Scanning electron microscopy (SEM), single fibre tensile testing (SFTT), FTIR analysis, Surface roughness, Thermogravimetric analysis (TGA), Differential Scanning Calorimetry (DSC), X-ray diffraction (XRD), ^{13}C (CP-MAS) NMR Spectroscopy

- **Chapter 5: Mechanical, morphological and thermal properties of ACB fiber reinforced epoxy composites**

This chapter deals with the characterization of raw acacia caesia bark fiber – epoxy composites, as regards their mechanical (tensile, flexural, Izod impact), hardness, SEM morphological and thermal (TGA, DSC) properties, as exposed in research discussion.

- **Chapter 6: Conclusions**

As far as the fibers are concerned, the following evidence has been elicited:

- The fiber diameter was very variable throughout the length, as typical of the structural unpredictability of bark fibers: the average value was calculated. The diameter of the fiber was reduced with gradual increase in alkali concentration during treatment.

- The density values of the Acacia caesia bark fibers are in the region of 1.2 g/cm^3, hence lower than common synthetic fibers, such as glass, carbon, or Kevlar. Alkali treatment, by removing non-cellulosic and non-structural components, leads to a slight increase in density, up to 1.26 g/cm^3 for a 15% NaOH solution treatment.
- The chemical properties showed that the hemicellulose (10.2%), ash (5.4%), moisture (10.4%), wax (0.1%) and other impurities were removed and the cellulose (44.1%), and lignin (28%) content was increased in the fibers with higher alkali concentration.
- FTIR study also confirmed by relevant peak intensities the partial removal of hemicellulose, lignin, wax, and other impurities from fiber surface. In addition, a strong hydrogen bond was noticed on treated fibers, which facilitated offering better mechanical properties to the fiber.
- The morphological study indicated that alkali treatment removed the surface impurities from the fibers. Also, the surface area of the alkali treated fibers improved, leading to better interfacial characteristics for the fiber in composites.
- The AFM illustrated the increase of fibers' surface roughness with gradual increase of alkali concentrations. The rougher fiber surface obtained, with an average roughness increased by a few microns, led to better fiber matrix interface and bonding and preserved the ratio between RMS roughness and average one into acceptable limits.
- Thermal analysis reveals that maximum degradation that is responsible for cellulose decomposition occurs at 308 °C on untreated fibers, while for alkali treated ones, fiber degradation may occur up to 326 °C, though no consistency is exactly reproducible for different levels of treatment. A confirmed result is that a higher level of residue, up to around 30% at 600 °C was obtained whatever the alkali concentration. As a whole, the thermal stability of the fibers was definitely enhanced by alkali treatments, as revealed by decomposition temperature and final residue, obtained by TGA/DSC plots of untreated and alkali-treated fibers.
- XRD analyses of the raw and treated ACBF portrayed this fiber as semi-crystalline (crystallinity index not exceeding 25%), not much increasing with alkali treatment.

For composites, the main evidences were:

- The maximum value of tensile strength (34.41 MPa), modulus (2.54 GPa), and elongation to break (3.23%), flexural strength (51 MPa) and modulus (2.83 GPa), impact energy (10.76 kJ/m^2) and Shore D hardness of 82.4. Best performance was obtained for 35 wt.% 20 mm long fibers.

- SEM fractography highlighted a strong fiber-matrix interfacial bond and adhesion.
- The highest thermal stability was observed by TGA and DSC on 35 wt% and 20 mm long fiber composites, showing that most of the degradation process occurred between 375 and 460 °C, with a residue equal to around 17% at 625 °C.

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List of Publications

1. **Sivasubramanian Palanisamy**, Mayandi Kalimuthu, Murugesan Palaniappan, Azeez Alavudeen, Nagarajan Rajini, Carlo Santulli, Faruq Mohammad & Hamad Al-Lohedan, Characterization of Acacia caesia Bark Fibers (ACBFs), *Journal of Natural Fibers,* (2021), 10241-10252.

2. **Sivasubramanian P**, Kalimuthu M, Palaniappan M, Alavudeen A, Rajini N, Santulli C, Effect of alkali treatment on the properties of acacia caesia bark fibres, *Fibers,* **9** (8) (2021).

3. **Sivasubramanian Palanisamy,** Mayandi Kalimuthu, Murugesan Palaniappan, Azeez Alavudeen, Nagarajan Rajini, and Carlo Santulli, Morphological characterization of soapbark fibers, *Journal of Materials Science Research and Reviews,* **8** (1) (2021), 19-26.

4. **Sivasubramanian Palanisamy**, Kalimuthu Mayandi, Shanmugam Dharmalingam, Nagarajan Rajini, Carlo Santulli, Faruq Mohammad & Hamad A. Al-Lohedan, Tensile properties and fracture morphology of Acacia Caesia bark fibers treated with different alkali concentrations, *Journal of Natural Fibers,* **19** (15) (2022), 11258 – 11269.

5. **Sivasubramanian Palanisamy**, Mayandi Kalimuthu, Rajini Nagarajan, José Maria Fernandes Marlet and Carlo Santulli, Physical, Chemical, and Mechanical Characterization of Natural Bark Fibers (NBFs) Reinforced Polymer Composites: A Bibliographic Review, *Fibers,* **11** (2), (2023).

List of Conferences attended

1. **Sivasubramanian P**, Mayandi K, Murugesan P, A Alavudeen, N Rajini, Carlo Santulli (2020), "Scanning Electron Microscopy morphological analysis of Soapbark fibres "*International Web Conference on Recent Trends and Developments in Applied Research and Industrial Practices (ICRTDARIP)*", Amity University, Kolkata. 20th - 21st November 2020.
2. **Sivasubramanian Palanisamy**, Mayandi Kalimuthu, Murugesan Palaniappan, Azeez Alavudeen, Nagarajan Rajini, Carlo Santulli (2021), "Characterization of Acacia Caesia Bark Fibres (ACBF) "*5th International Conference on Natural Fibers (ICNF21)",* University of Minho, Guimaraes, Portugal. 17th - 19th May 2021.